Submission ID #: 61026
Scriptwriter Name: Bridget Colvin
Project Page Link: http://www.jove.com/files_upload.php?src=18612978

Title: In Situ Transmission Electron Microscopy with Biasing and Fabrication of Asymmetric Crossbars Based on Mixed-Phased alpha-VO_x

Authors and Affiliations: Shruti Nirantar[1,*], Edwin Mayes[2,*], and Sharath Sriram[1]
*These authors equally contributed to the work

[1]Functional Materials and Microsystems Research Group and the Micro Nano Research Facility, RMIT University
[2]RMIT Microscopy and Microanalysis Facility, RMIT University

Corresponding Author:
Shruti Nirantar
shruti.nirantar@gmail.com

Co-authors:
edwin.mayes@rmit.edu.au
sharath.sriram@rmit.edu.au

Author Questionnaire

1. Microscopy: Does your protocol involve video microscopy, such as filming a complex dissection or microinjection technique? **N**

2. Software: Does the part of your protocol being filmed demonstrate software usage? **Yes**

3. Filming location: Will the filming need to take place in multiple locations (greater than walking distance)? **N**

Script Length
Number of shots: **43**

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Shruti Nirantar**: This protocol can help improve our understanding of the nanostructural changes that occur within an alpha-VO_x-based cross-point device when the changes are biased in situ **[1]**.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

REQUIRED:

1.2. **Edwin Mayes**: This protocol allows the visual analysis of nanostructural changes that relate to the device biasing in real-time and can be used on any metal-insulator-metal stacked structure compatible with high vacuum conditions **[1]**.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

OPTIONAL:

1.3. **Shruti Nirantar**: This protocol can be easily extended to performing in situ temperature and biasing analyses to reveal nanostructural changes down to the atomic level **[1]**.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

Protocol

2. Fabrication Process and Electrical Characterization

2.1. To pattern the bottom of the electrode of the cross-point device with photoresist, first spin coat the photoresist onto the wafer at 3000 revolutions per minute **[1]** before soft baking at 95 degrees Celsius for 60 seconds **[2]**.

2.1.1. WIDE: Talent spin coating device

2.1.2. Talent placing device at 95 °C

2.2. Next, use a 405-nanometer laser to expose the device to 25 millijoules/square-centimeter **[1]** and bake at 120 degrees Celsius for 120 seconds **[2]**.

2.2.1. Talent applying laser to device

2.2.2. Talent placing device at 120 °C

2.3. Use a 400-nanometer laser to flood expose the device with 21 milliwatts/square-centimeter **[1]** and use developer to develop the pattern **[2]**.

2.3.1. Talent applying laser to device

2.3.2. Talent placing device into developer, with developer visible in frame

2.4. Rinse the device with deionized water **[1]** and use a physical vapour deposition system to deposit 10 nanometers of titanium and 100 nanometers of platinum onto the substrate pattern on layer 1 **[1]**.

2.4.1. Talent rinsing device

2.4.2. Metal(s) being deposited From Authors: This step has long waiting time, hence not captured in video. Providing some still images for this process, either use that or it is fine to just say the metals were deposited. This is a very standard process and not crucial for the novelty here

2.5. To lift-off the deposited metals, place the substrate in an acetone bath for approximately 20 minutes **[1]** before applying ultrasonic vibrations for 2 minutes **[2]** and rinsing with acetone and isopropyl alcohol **[3-TXT]** and repeat the process till the clean patterned wafers are achieved **[4]**.

2.5.1. Talent placing substrate into bath, with acetone container visible in frame

2.5.2. Substrate being vibrated

2.5.3. Talent placing substrate into bath, with acetone and IPA containers visible in frame **TEXT: Repeat if lift-off not clean**

2.5.4. Note from Authors: Capture the video as well as photographs of the wafers patterned with bottom-electrode (Layer 1)

2.6. Repeat the same photolithography or patterning process to patter the second layer of amorphous vanadium oxide and the third layer onto the top of the electrode with 20-nanometer titanium and 200-nanometer platinum **[1]**.

2.6.1. Note from Authors: Showing the final patterned sample in video and photographs

3. Biasing Chip Mounting on Gridbar

3.1. For mounting of the biasing chip, first clean the chip in a glass Petri dish filled with acetone with gentle rotation for 2 minutes **[1]**.

3.1.1. WIDE: Talent placing chip into dish on rotator, with acetone container visible in frame

3.2. After washing, transfer the chip into a Petri dish filled with methanol for an additional two minutes of rotation **[1]** before blowing the chip dry with low pressured nitrogen **[2]**.

3.2.1. Shot of chip rotating in dish, with methanol container visible in frame

3.2.2. Chip being blown dry

3.3. Align the dried biasing chip in square trenches of gridbar **[1-TXT]** and use screws to fix the grid cover onto the biasing chip, finalizing the placement of the E-chip on the gridbar **[2]**.

3.3.1. Chip being aligned *Videographer: Important step* **TEXT: See text for gridbar fabrication details**

3.3.2. Gridbar being screwed onto chip *Videographer: Important step*

4. Lamella Preparation and biasing chip mounting

4.1. To mount the biasing chip with lamella, use conductive carbon tape to mount the newly prepared sample onto a metal stub **[1]** and load the stub into a focus ion beam chamber **[2]**.

4.1.1. WIDE: Talent mounting sample onto stub

4.1.2. Talent loading stub info FIB chamber

4.2. Apply additional tape on the sample for grounding to avoid charging issues **[1]** and load the biasing chip-mounted gridbar in the chamber at a 52-degree angle **[2]**.

4.2.1. Tape being applied

4.2.2. Chip being mounted at angle

4.3. Use the microscope physical control panel and software to focus, astigmate, and align the electron beam on a sample surface **[1]** and check the eucentric height of the focused sample location and beam coincidence for the electron and ion beams **[2]**.

4.3.1. Talent at computer, focussing and/or astigmatizing, and/or aligning beam, with monitor visible in frame

4.3.2. SCREEN: section 4 – 1: 02:08-02:30 *Video Editor: please speed up*

4.4. Click the **Auto TEM** program to run the program on the focused sample location **[1]** and use silicone milling to create cross fidicuial alignment markers **[2]**.

4.4.1. SCREEN: section 4 – 1: 03:20-04:36 *Video Editor: please speed up*

4.4.2. SCREEN: section 4 – 2: 00:00-00:18 *Video Editor: please speed up*

4.5. Deposit a 1.5-micrometer-thick carbon protective layer over the 20- x 5-micrometer area between the alignment markers and use a 5-nanoangstrom beam current to mill trenches on either side of the carbon protective layer **[1]**.

4.5.1. SCREEN: section 4 – 2: 06:05-28:44 *Video Editor: please speed up*

4.6. Thin the lamella with subsequent 1-nanoangstrom and a 300 picoangstrom ion beam currents to reach a 1-micrometer thickness **[1]**.

4.6.1. SCREEN: section 4 – 3: 03:54-04:45 *Video Editor: please speed up*

4.7. Tilt the sample to 7 degrees to perform a J-cut on the lamella for separation from the substrate **[1]**.

4.7.1. Talent using knobs to tilt sample/perform cut

4.8. To attach the lamella to the manipulator needle, tilt the sample to 0 degrees and use platinum to attach the lamella to the needle **[1]**.

4.8.1. SCREEN: section 4 – 3: 08:53-09:37 *Video Editor: please speed up*

4.9. After attaching to the micromanipulator, use a final cut to separate the lamella **[1]** from the substrate and slowly retract the micromanipulator **[2]**.

4.9.1. SCREEN: section 4 - 3: 11:00-14:40 *Video Editor: please speed up*

4.9.2. SCREEN: section 4 – 3: 16:15-16:35

4.10. Focus the beam on the top edge of biasing chip on the gridbar and use the needle to bring the lamella slowly toward the biasing chip **[1]**.

4.10.1. SCREEN: section 4 – 3: 20:25-21:24 *Video Editor: please speed up*

4.11. Align the lamella in the center of the 17-micrometer gap on the top edge of the biasing chip **[1]** and slowly move the gap down until it barely touches the chip surface **[2]**.

4.11.1. Talent aligning beams with knobs, with monitor visible in frame *Videographer: Important step*

4.11.2. Gap being moved until barely touching chip surface *Videographer: Important step*

4.11.3. Note from Authors: Screen captures for 4.11 will be provided by the author

4.12. Use platinum to weld the bottom edges of the lamella to the chip and use silicon milling to cut the micromanipulator free from the lamella **[1]**.

4.12.1. SCREEN: section 4 – 3: 22:00-27:23 *Video Editor: please speed up*

4.13. Retract the micromanipulator **[1]** and connect the top edges of the lamella with platinum traces to the two electrodes of the biasing chip **[2]**.

4.13.1. SCREEN: section 4 – 3: 27:23-27:28

4.13.2. SCREEN: section 4 – 3: 28:20-39:04 *Video Editor: please speed up*

4.14. Tilting the specimen front and back by 2 degrees to ensure parallel faces and a uniform thickness, use a consecutive 300-picoangstrom and 100-picoangstrom beam to thin the center region of lamella to less than 100-nanometers thick **[1]**.

4.14.1. SCREEN: section 4 – 3: 41:58-01:03:37 *Video Editor: please speed up*

4.15. Polish out the ion beam-damaged layer with the gallium beam accelerating voltage of 5 kilovolts at a 5-degree angle to the surface on both faces and use isolation cuts in the thinned region to remove the short connection between the top and bottom electrodes of the device to create a current path from bottom to the top electrode through the active region **[1]**.

4.15.1. SCREEN: f200 TEM: 15:45-16:00 *Video Editor: please speed up*

4.16. Then mount the biasing chip with lamella on the biasing chip holder **[1]** and load the biasing chip holder into the transmission electron microscopy chamber **[2]**.

4.16.1. Chip being mounted onto in situ biasing holder

4.16.2. Talent placing holder into TEM chamber

5. In Situ Transmission Electron Microscopy (TEM)

5.1. For in situ transmission electron microscopy imaging, carefully connect the wires from the biasing chip holder to the source meter and a control computer **[1]**.

5.1.1. WIDE: Talent connecting wire(s)

5.1.2. Talent checking chamber pressure

5.2. When the transmission electron microscopy chamber pressure drops to desired value, use the transmission electron microscopy control knobs to focus, astigmate, and align the electron beam on a cross section of the lamella surface **[1]** and apply voltage sweeps **[2-TXT]**, collecting the transmission electron microscopy micrographs in situ **[3]**.

5.2.1. Talent aligning beams with knobs, with monitor visible in frame

5.2.2. SCREEN: TEM in situ bias: 21:00-21:30 *Video Editor: please speed up* **TEXT: Alternative: apply constant voltage at different biasing voltages**

5.2.3. LAB MEDIA: Figure 8a

5.3. In situ nanostructural changes occurring within the lamella on the application of bias can then be observed **[1]**.

5.3.1. SCREEN: TEM in situ bias: 21:30-22:30 *Video Editor: please speed up*

Protocol Script Questions

A. Which steps from the protocol are the most important for viewers to see? Please list 4 to 6 individual steps.

3.3., 4.11., 5.2.

B. What is the single most difficult aspect of this procedure and what do you do to ensure success? Please list 1 or 2 individual steps from the script above.
4.15. is a last and critical step in lamella preparation, where is it very hard to locate positions where separation cuts are required. With the use of STEM detectors a few tens of nanometer layers can also be seen clearly. We use this detector to locate the exact positions before making isolation cuts.

Results

6. Results: Representative In Situ Electrical TEM

6.1. In this image, a representative transmission electron microscopy micrograph of the intact lamella can be observed **[1]**. The diffraction patterns in the inset indicate the amorphous nature of the oxide film **[2]**.

6.1.1. LAB MEDIA: Figure 8A
6.1.2. LAB MEDIA: Figure 8A *Video Editor: please emphasize inset image*

6.2. At 4 volts, a localized crystalline region forms in the oxide layer **[1]**. In this analysis, *d*-spacing was 0.35 nanometers **[2]**, corresponding to the 011 plane of the vanadium oxide-M1 phase **[3]**.

6.2.1. LAB MEDIA: Figure 8B *Video Editor: please trace/emphasize yellow dashed rectangle*
6.2.2. LAB MEDIA: Figure 8B *Video Editor: please emphasize bottom right inset*
6.2.3. LAB MEDIA: Figure 8B *Video Editor: please emphasize top left inset*

6.3. At 5 volts, multiple localized crystal islands oriented in different directions with respect to the substrate can be observed within the oxide **[1]**.

6.3.1. LAB MEDIA: Figure 8C *Video Editor: please trace/emphasize yellow dashed shapes*

6.4. A *d*-spacing of 0.27 nanometers corresponding to the vanadium oxide-A phase **[1]** and a *d*-spacing of 0.26 nanometers corresponding to the vanadium oxide-M1 phase are apparent **[2]**.

6.4.1. LAB MEDIA: Figure 8C *Video Editor: please emphasize bottom right inset*
6.4.2. LAB MEDIA: Figure 8C *Video Editor: please top left inset*

6.5. At 6 volts, Moiré fringes can be observed, with multiple nucleation sites apparent within the lamella **[1]**.

6.5.1. LAB MEDIA: Figure 8D *Video Editor: please trace/emphasize yellow dashed shapes*

6.6. At this voltage, evidence of the different orientations of the vanadium oxide-M1 crystal islands can also be noted **[1]**.

6.6.1. LAB MEDIA: Figure 8D *Video Editor: please emphasize bottom right and top left insets*

Conclusion

7. Conclusion Interview Statements

7.1. **Shruti Nirantar**: It is important to remember to perform the titanium capping, as without it, the device fabrication will fail **[1]**.

7.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera (2.6.)

7.2. **Edwin Mayes**: Using this method, diffraction pattern, electron diffraction X-ray spectroscopy, and electron energy loss spectroscopy mapping data can also be collected at different biasing voltages in situ **[1]**.

7.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera